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FORM PTO-1390 (REV. 9-2001)	US DEPARTMENT OF COMMERC	E PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER				
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371			J-1950 U.S. APPLICATION NO (If known, see 37 CFR 15  10/030604				
INTERNATIONA		TERNATIONAL FILING DATE	PRIORITY DATE CLAIMED				
PCT/JP99/03942		July 1999 (22.07.99)	22 July 1999 (22.07.99)				
AQUEOUS FLOOR POLISHING COMPOSITION							
	OR DO/EO/US KONDO,						
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:							
1. X This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.							
2. This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.							
This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.							
4. The US ha	4. The US has been elected by the expiration of 19 months from the priority date (Article 31).  5. A copy of the International Application as filed (35 U.S.C. 371(c)(2))						
$\begin{bmatrix} 5 & 1 \\ & a \end{bmatrix}$ A copy of $\begin{bmatrix} 1 \\ & a \end{bmatrix}$	s attached hereto (required on	ly if not communicated by the Internation	nal Bureau).				
b. 🗍 1	nas been communicated by the	e International Bureau.					
c. 🔲 i		ion was filed in the United States Received					
		nternational Application as filed (35 U.S	.C. 371(c)(2)).				
	s attached hereto.	Lunder 35 II S.C. 154(d)(4)	_				
b. has been previously submitted under 35 U.S.C. 154(d)(4).  7. Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))							
a.	——————————————————————————————————————						
	. D. I. I amministed by the International Bureau						
c. have not been made; however, the time limit for making such amendments has NOT expired.							
d. 🔲							
8. X An Englis	h language translation of the	amendments to the claims under PCT Art	ticle 19 (35 U.S.C. 371 (c)(3)).				
9. An oath o	r declaration of the inventor(s	) (35 U.S.C. 371(c)(4)).					
10. X An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).							
Items 11 to 20	) below concern document(s	) or information included:	,				
		t under 37 CFR 1.97 and 1.98.					
12. An assi	An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.						
13. A FIRS	T preliminary amendment.						
14 A SEC							
15. A subst	15. A substitute specification.						
16. X A chan							
17. A comp							
18. A secon	18. A second copy of the published international application under 35 U.S.C. 154(d)(4).						
19. A seco	19. A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).						
20. X Other items or information: See Attachment 1							
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U OINERNATIONAL APPLICATION NO ATTORNEY'S DOCKET NUMBER J-1950 PCT/JP99/03942 CALCULATIONS PTO USE ONLY 21. X The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO. .... \$1040.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO . \$890.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO . . . . . . . . . \$740.00 International preliminary examination fee (37 CFR 1.482) paid to USPTO \$710.00 but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00 ENTER APPROPRIATE BASIC FEE AMOUNT = 890.00 Surcharge of \$130.00 for furnishing the oath or declaration later than \$ months from the earliest claimed priority date (37 CFR 1.492(e)). 0.00 \$ NUMBER EXTRA **RATE** NUMBER FILED **CLAIMS** \$ 0.00 \$18.00 0 Total claims -20 =0.00 \$ \$84.00 Independent claims 2 -3 = '0.00 \$ \$280.00 MULTIPLE DEPENDENT CLAIM(S) (if applicable) \$ 890.00 TOTAL OF ABOVE CALCULATIONS = Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above 0.00 are reduced by 1/2. \$ 890.00 **SUBTOTAL** Processing fee of \$130.00 for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492(f)). 0.00 890.00 TOTAL NATIONAL FEE = \$ Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be \$ accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property 0.00 890.00 TOTAL FEES ENCLOSED Amount to be refunded: \$ charged: A check in the amount of \$ \_\_\_\_\_\_ to cover the above fees is enclosed. Please charge my Deposit Account No. 50-0231 in the amount of \$ 890.00 to cover the above fees. A duplicate copy of this sheet is enclosed. c. X The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 50-0231 . A duplicate copy of this sheet is enclosed. Fees are to be charged to a credit card. WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038. NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status. SEND ALL CORRESPONDENCE TO. Warren R. Bovee SIGNATURE S. C. Johnson Commercial Markets, Inc. Warren R. Bovee 8310 16th Street - M/S 509 NAME P.O. Box 902 Sturtevant, WI 53177-0902 26,434 **USA** REGISTRATION NUMBER

#### DESCRIPTION

Aqueous Floor Polishing Composition
Technical Field

The present invention relates to an aqueous floor polishing composition excellent in gloss restorability. In particular, the present invention relates to an aqueous floor polishing composition which is desirable for the application to floor surface of wooden floor material, floor material comprised of synthetic resin, or stone floor made of concrete or marble, especially, for dry maintenance which is carried out as daily maintenance by using a combination of cleaning with an automatic floor cleaning machine and buffing with a high-speed polisher.

# 15 Background Art

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Floor polishing agents have been used for the floor surface of wooden floor material, chemical floor material made of synthetic resin, or stone floor material comprised of concrete or marble for the purpose of keeping the beauties of the floor material and protecting the floor surface. The floor polishing agents generally include oil-type agents using solvents, aqueous agents, emulsion-type agents and the like. For example, Japanese Patent Publication Nos. Sho 47-14019 and Sho 47-15597 disclose floor polishing compositions which use a polyvalent metal compound in a polymer of an ethylenically unsaturated compound. The

coating film obtained by applying and drying this floor polishing composition is excellent in durability or travelling, and nowadays, this type of floor polishing composition is dominantly used.

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A floor polishing agent containing an aqueous emulsion of polyurethane resin has been developed for the purpose of improving anti-slip properties. e.g., Japanese Patent Publication No. Sho 53-22548. addition, Japanese Patent Application laid-Open No. Sho 61-148273 discloses that in order to improve heel-mark resistance or scuff resistance, the film durability can be enhanced by using a mixture of an aqueous resin with colloidal silica and butoxyethyl phosphate in combination. Furthermore, Japanese Patent Application Laid-Open No. Sho 8-41382 discloses a method for improving film durability. However, in the aforementioned prior art, there is no disclosure of significantly improving gloss restorability by buffing, and hence, the development of new technique has been desired in this field.

The buffing operations in dry maintenance are carried out for restoring the floor surface by plastically deforming or cut-removing small damages on the coating surface caused by walking, etc. and restoring gloss lowered by walking, etc.

In general, the improvement in the gloss restorability by buffing with floor polish may be

achieved by adjusting a composition of resin in an acryl-styrene type emulsion which is a main component of a polishing agent, but heel-mark resistance, scuff resistance, etc. are deteriorated. Accordingly, it has been very hard to obtain a floor polishing composition which is excellent in gloss restorability by buffing, and besides, excellent in heel mark resistance and scuff resistance.

### Disclosure of Invention

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The present invention is to provide a novel,

aqueous floor polishing composition capable of forming
a coat showing excellent gloss restoration by buffing
while retaining general properties such as durability
required for floor polishing agents.

The inventors found out, as a result of eager investigation and study, that the use of a butadiene type copolymer component in an aqueous floor polishing resin composition will readily afford the evenness of a coat after buffing by means of a high-speed polisher, thereby improving the gloss restoration by buffing. Thus, according to the present invention, there is provided an aqueous floor polishing composition characterized by containing 5 to 100 parts as solid of a styrene-butadiene type copolymer emulsion based on 100 parts as solid of an acryl-styrene type resin emulsion.

The styrene-butadiene type copolymer emulsion

which can be used for the present invention is preferably that obtained by copolymerization of styrene and butadiene. Any type of thus obtained copolymer may be used regardless of molecular weight, molecular 5 structure and preparation method. For the copolymerization of styrene and butadiene aromatic and/or aliphatic monomers such as methyl methacrylate and the like may be incorporated at an arbitrary copolymerization ratio. Copolymerization with 10 unsaturated fatty acid such as acrylic acid and methacrylic acid or dispersion into water using emulsifiers may be conducted for water dispersibility or water solubility. Such butadiene type copolymer latex, including copolymers obtained by emulsion 15 copolymerization of butadiene and a variety of monomers, is widely used for compositions for paper coating or the like. A typical example of such latex is described in Japanese Patent Publication No. 54-6575. The water base floor polishing agent of the 20 present invention can be prepared by adding such butadiene type copolymer latex into acryl-styrene type resin emulsions. It can be also prepared by polymerizing monomers containing butadiene in the presence of acryl-styrene type copolymerization 25 components or polymerizing monomers such as aliphatic unsaturated compounds and styrene in the presence of butadiene type copolymerization components. Concerning

the ratio of acryl-styrene type resin to styrene-butadiene type copolymer emulsion, the styrene-butadiene type copolymer emulsion is in the range of 5 to 100 parts, preferably in the range of 5 to 50 parts, with respect to 100 parts of acryl-styrene type resin. The butadiene in the styrene-butadiene type copolymer emulsion is preferably in the range of 25 to 70 % by weight, and the styrene in the styrene-butadiene type copolymer emulsion is preferably in the range of 0 to 75 % by weight, and more preferably in the range of 30 to 75 % by weight.

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An example of the acryl-styrene type resin emulsion which can be preferably used in the present invention is the resin composition disclosed in Japanese Patent Publication No. 44-24409 or the like. The composition comprises a copolymer of alpha, betaethylenic unsaturated carbonic acids and ester derivatives thereof, especially acrylic acid or methacrylic acid and ester derivatives thereof, and aromatic monomers such as styrene. Specifically an ethylenic unsaturated compound is selected from the group consisting of styrene, methylstyrene, acrylic acid, methyl acrylate, ethyl acrylate, propyl acrylate, butyl acrylate, hexyl acrylate, 2-ethylhexyl acrylate, lauryl acrylate, methacrylic acid, methyl methacrylate, ethyl methacrylate, propyl methacrylate, butyl methacrylate, hexyl methacrylate, 2-ethylhexyl

methacrylate, vinyl acetate, acrylonitrile, itaconic acid, maleic acid and the like, thereby obtaining the polymer emulsion by a known emulsion polymerization method.

In the present invention, for the purpose of 5 satisfying various characteristics required for the floor polishing agent, various additives can be contained in the aqueous floor polishing composition. Among the characteristics, an appropriate sliding 10 property can be obtained and its black heel mark resistance and durability are made excellent by adding a wax to the composition. As the wax, it is possible to use an aqueous solution of a natural wax or a synthetic wax dispersed therein which is generally used 15 for floor polishing. Specifically, the natural wax includes carnauba wax, paraffin wax and the like. synthetic wax includes polyethylene, polypropylene and oxides thereof as well as waxs obtained by polymerizing unsaturated monomers such as ethylene, propylene and 20 the like or monomers selected from the group consisting of  $\alpha$ - or  $\beta$ -ethylenically unsaturated carboxylic acid, alkyl esters thereof and the like. Though the wax content in the aqueous floor polishing composition of the present invention is optionally determined, 0 to 20 25 % by weight is preferable.

A crosslinking agent is added to the aqueous floor polishing composition of the present invention to

improve the duralibility of an obtained coating film.

The crosslinking used in the present invention includes polyvalent metal salts or polyvalent metal complexes by which a metal crosslinking can be formed.

Specifically, it is possible to use calcium, magnesium, zinc, barium, aluminum, zirconium, nickel, iron, cadmium, strontium, bismuth, beryllium, cobalt, lead, copper, titanium and antimony. In particular, calcium, magnesium, zinc and aluminum are preferable. The

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ligand for forming the polyvalent metal complexes includes carbonate ion, acetate ion, oxalate ion, malate ion, hydroxyacetate ion, tartrate ion, acrylate ion, lactate ion, octate ion, formate ion, salicylate ion, benzoate ion, gluconate ion, glutamate ion, and glycine, alanine, ammonia, morpholine, ethylenediamine, dimethylaminoethanol, diethyamionethanol,

well as inorganic acids, organic acids, amino acids, amines and the like which are similar thereto. In particular, zinc ammonium carbonate, calcium ethylenediamine-ammonium carbonate, zinc ammonium acetate, zinc ammonium acrylate, zinc ammonium malate, zirconium ammonium malate, zic ammonium aminoacetate, calcium ammonium alanine and the like are preferable.

monoethanolamine, diethnolamine, triethanolamine, as

In the aqueous floor polishing composition of the invention, by incorporating an alkali-soluble resin, it is possible to improve the leveling property,

peelability and glossiness. As the alkali-soluble resin, there can be included styrene-maleic acid copolymer resin, rosin-maleic acid copolymer resin, water-soluble acrylic resin, water-soluble polyester resin, water-soluble epoxy resin, or the like. The content of the alkali-soluble resin in the aqueous floor polish composition of the present invention can arbitrarily be determined.

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In the aqueous floor polishing composition of the invention, by incorporating an aqueous polyurethane type resin, it is possible to improve the durability. As the aqueous polyurethane type resin, there can preferably be used one containing a carboxylic acid and/or carboxylate bonded to a chain of a polyurethane type resin. Such a resin can be obtained by, for example, when producing a polyurethane type resin, adding diol or the like having a carboxylic acid group to diol and di-isocyanate, neutralizing the carboxylic acid group as needed, and effecting polymerization. The introduction of a carboxylic acid group affords an aqueous polyurethane type resin having waterdispersibility or water-solubility. Further, adding an emulsifying agent can make the resin aqueous as needed.

The aqueous floor polishing composition of the invention may contain an plasticizer or film-forming assistant. As the plasticizer, there are included dibutyl phthalate, dioctyl phthalate, 2-pyrrolidone,

octyl diphenyl phosphate, tributoxyethyl phosphate, and the like. As the film-forming assistant, there are preferably used alkylene glycol monoalkyl ether, dialkylene glycol monoalkyl ether, trialkylene glycol monoalkyl ether, and there are specifically included diethylene glycol monobutyl ether, diethylene glycol monoethyl ether, dipropylene glycol monomethyl ether, and the like.

The aqueous floor polishing composition of the invention may contain silica sol. Further, the composition may contain a fluorine type surfactant or a preservative.

The present invention will be described below in great detail by giving Examples and Comparative Example which will illustrate the technical effects of the present composition.

# EXAMPLE 1

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Ten (10) parts of a styrene-butadiene type copolymer latex composed chiefly of styrene/butadiene = 60/40, having Tg of -1°C was added to 100 parts of an acryl-styrene type resin emulsion composed of butyl acrylate/methyl methacrylate/ styrene/methacrylic acid = 32/28/20/20, having Tg of 46°C and an acid value of 130 to prepare an aqueous floor polishing composition 1 of the present invention.

# EXAMPLE 2

An aqueous floor polishing composition 2 of the

present invention was prepared in the same manner as in Example 1 except that the amount of the styrene-butadiene type copolymer latex was changed to 20 parts.

#### EXAMPLE 3

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An aqueous floor polishing composition 3 of the present invention was prepared in the same manner as in Example 1 except that the amount of the styrene-butadiene type copolymer latex was changed to 30 parts.

# COMPARATIVE EXAMPLE

Only the acryl-styrene type resin emulsion used in Example 1 was used and made an aqueous floor polishing composition 4.

The aqueous floor polishing composition obtained in Example 2 was dried at 60°C for 12 hours to prepare a sample for analysis, which was then analyzed as to the butadiene contained in the composition by using an infrared spectrophotometer (KBr tablet method; integration for 100 times using 60SX Fourier transform infrared spectrophotometer manufactured by Nicolet). As a result, absorption peaks were observed in the wavelength regions of 960 to 970 cm<sup>-1</sup> (trans 1,4 CH bending vibration), 900 to 920 cm<sup>-1</sup> (R-CH=CH bending vibration) and 1640 cm<sup>-1</sup> (C=C stretching vibration) which are the characteristic absorption bands of butadiene copolymer. Further, as a result of another analysis which was carried out by using a thermal

decomposition gas chromatograph (HP5890A gas

chromatograph manufactured by Hewlett Packard Co.,
Ltd.; FID detection; high frequency furnace type
thermally decomposing apparatus manufactured by Nippon
Bunseki Kogyo K.K.; thermal decomposition at 590°C for
5 sec.), the butadiene monomer decomposed was
confirmed, so that it was confirmed that butadiene was
contained as the monomer component in the aqueous floor
polishing composition of the present invention.

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The aqueous floor polish compositions 1 - 4

prepared in Examples 1-3 and Comparative Example 1 were

used to prepare floor polishing agents. To 100 parts

by solid weight of the composition, 10 parts of

tributoxyehtyl phosphate (a plasticizer), 25 parts of

diethylene glycol monoethyl ether (a filming aid), 0.05

parts of fluorine-based detergent Zonyl FSJ (25%

solid)(a product of Du pont), 6.37 parts of ammonium

carbonate zinc aqueous solution (12% solid), 6.25 parts

of styrene maleic acid resin SMA-2625A (15% solid)(a

product of ATOCHEM), 18.76 parts of polyethylene wax

emulsion Hytec E-4B (40% solid) (a product of Toho

Kagaku Inc.), and 0.68 parts of Deltop (a preservative,

a product of Takeda Chemical Industries) are added

successively.

With each aqueous floor polishing agent thus obtained, gloss restoration by buffing was determined.

The results are shown in Table 1. The determination method is as follows:

- Glossiness: determined according to JIS K-3920 (Test method for floor polish);
- 2. Gloss restoration by buffing: Onto a homogeneous vinyl floor tile (a product of TORI Inc., product name: MATICO S PLAIN No.5626), the sample polishing agent was applied five times, and then the glossiness was measured (a). After that, the gloss was removed by using an automatic floor washing machine J-CRUISE (a product of Johnson Professional Inc.)((b)), then the tile was buffed twice by using Ultra high speed polisher SPRINT-2000BP(a product of Johnson Professional Inc.) with a green pad (c and d), to determine the gloss restoration by buffing from the glossiness difference before and after buffing.

Table 1
Glossiness Restoration by Buffing

		Examples		Comp.Example	
	1	2	3	4	
Glossiness					
(a) 5x application	73	72	70	75	
(b) before buffing	51	50	52	52	
(c) after first					
buffing	65	65	67	58	
(d) after second					
buffing	67	69	70	60	
Gloss restoration(%)					
after 1st buffing	64	68	83	26	
before 2nd buffing 73		86	100	35	

Gloss restoration(%)=(gloss after buffing - gloss before
buffing)/(gloss after 5x application - gloss before
buffing)

# Effect of Invention

As shown in Table 1, high rates of gloss restoration are attained by using the composition of the invention. In other words, the use of an aqueous floor polishing composition containing 5 to 100 parts as solid of a styrene-butadiene type copolymer emulsion based on 100 parts as solid of an acryl-styrene type resin emulsion, according to the invention, will afford both high gloss

restorability in buffing and high durability that have been long desired as properties for floor polishing agents. The aqueous floor polishing composition of the invention is particularly suitable for 'dry maintenance' that is a daily maintenance work comprised of washing by means of an automatic washer and buffing by means of a high-speed polisher.

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#### CLAIMS

- An aqueous floor polishing composition comprising 1. 5 to 100 parts by weight of a solid styrene-butadiene type copolymer emulsion based on 100 parts solids of an acrylicstyrene type resin emulsion.
- A method of imparting gloss to a flooring surface 2. and maintaining said gloss comprising:
- a) applying to a floor an aqueous floor polishing composition containing 5 to 100 parts by weight of a solid styrene-butadiene type copolymer emulsion based on 100 parts solids of an acrylic-styrene type resin emulsion;
- b) allowing said composition to dry to form a glossy film on said floor; and
- c) periodically washing said film with water and thereafter buffing said film with a high-speed polisher to restore said gloss.

# ABSTRACT

An aqueous floor polishing composition contains 5 to 100 parts as solid of a styrene-butadiene type copolymer emulsion based on 100 parts as solid of an acryl-styrene type resin emulsion. The novel aqueous floor polishing composition forms a coat excellent in gloss restoration by buffing.

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J-1950

# COMBINED DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

As below named inventors, I hereby declare that:

This patent application is based on PCT International Application No. PCT/JP99/003942; assigned U.S. Application No. 10/030,604.

Our residence, post office address and citizenship are as stated below next to our names:

We believe we are the original, first and joint inventors of the subject matter which is claimed and for which a patent is sought in the invention entitled: AQUEOUS FLOOR POLISHING COMPOSITION, filed with the United States Receiving Office on 09 January 2002.

We hereby state that we have reviewed and understand the contents of the above identified specification, including the claims.

We acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

We hereby appoint Warren R. Bovee (Reg. No. 26,434), Neil E. Hamilton (Reg. No. 19,869) and Renee J. Rymarz (Reg. No. 35,666) as our attorneys to prosecute this application and to transact all business in the Patent and Trademark Office connected herewith.

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We hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patents issued thereon.

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